## ABSTRACT

The present invention provides an in-spray glow discharge ionization method and apparatus which can be used together or alternately with the most widely used ionization method of mass spectrometry, such as an atmospheric pressure chemical ionization (APCI) method or an electrospray ionization (ESI) method while enhancing ionization efficiency using a gas exhibiting Penning effect. The inspray glow discharge ionization apparatus has a supply port (A) supplying a fluid containing a compound to be measured, a gas blowing port (B) which surrounds the supply port (A) and which blows a gas exhibiting Penning effect to nebulize the fluid supplied from the supply port (A), a ground-side discharge electrode (E) provided at a generation port (C) at which the nebulized flow is generated, and a voltage application-side discharge electrode (F) which is disposed in the traveling direction of the nebulized flow and opposed to the ground-side discharge electrode. In this in-spray glow discharge ionization method, while the fluid is nebulized by a spray gas (1), components of the compound to be measured which constitutes the fluid are ionized by the excited spray gas (1) exhibiting Penning effect, so that measurement is performed by a mass spectrometer.